Nanogeology of Metamorphic Magnetite using Three-Dimensional Atom Probe and Focused Ion Beam Secondary Ion Mass Spectroscopy

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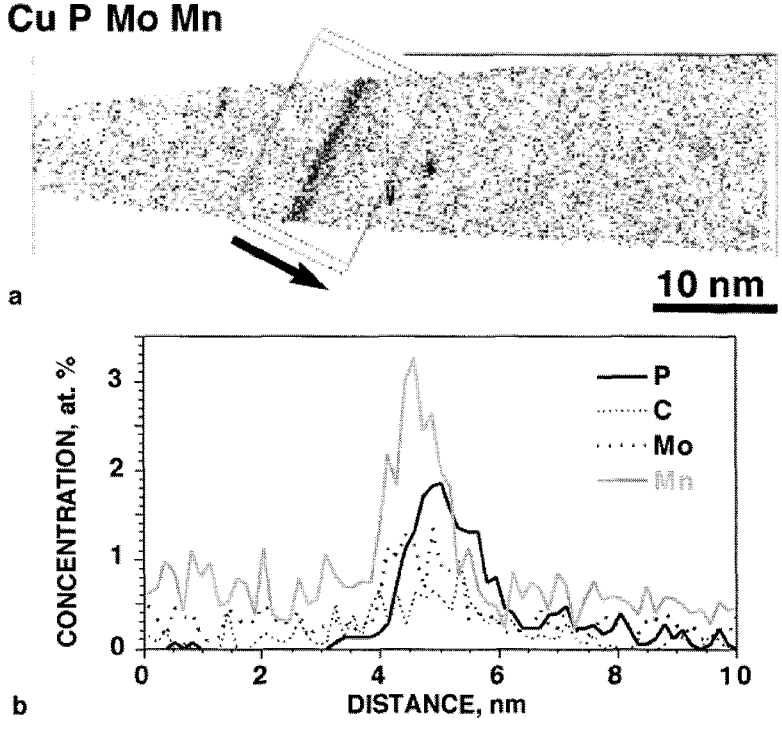
Objectives of this work

- A 3D near-atomic scale elemental map of a geological sample.
- Demonstrate the planetary science potential of the Local Electrode Atom Probe (LEAP).
  - Potential of LEAP analysis for non-conductive samples:
    - Terrestrial geology and geomicrobiology,
    - Apollo samples from the Moon,
    - Samples returned by the Stardust, Genesis and Mars missions.
- Demonstrate the potential of the Mini-LEAP for the *in-situ* analysis of planetary materials.
  - NASA is currently developing a prototype Mini-LEAP at JPL.

Schematic Illustration of APFIM Analysis

From Miller, et al. (1996) Atom Probe Field Ion Microscopy Oxford University Press

Analytical Capability of 3DAP



Atom probe analysis in the vicinity of an interface in a pressure vessel steel. Enrichments of P, C, Mo and Mn are evident.

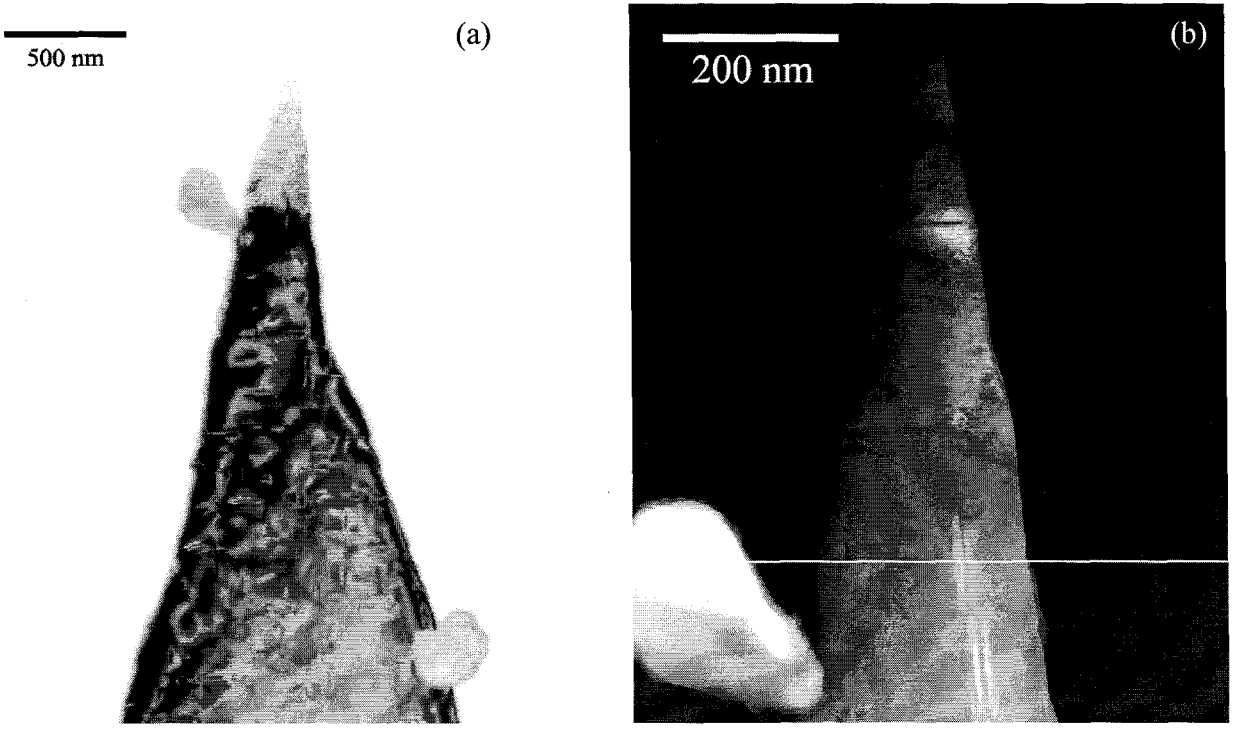
From Miller (2000) Atom Probe Tomography : Analysis at the Atomic Level, Plenum Publishing Corporation.

Why Magnetite?

- A common mineral on Earth and Mars.
- One of the more conductive minerals.
  - Resistivity = 52 x 10^{-4} ohm-cm.
- This particular magnetite contains disk-shaped exsolutions approx. 40 nm in diameter, 1-3 nm thick and about 10^4 platelets/μm^3.
- EDS shows Mn and Al concentrated in these precipitates.
- Quantitative analysis has been limited by the thickness of this second phase.

Atom Probe is a technique that can potentially quantify the composition of these precipitates.

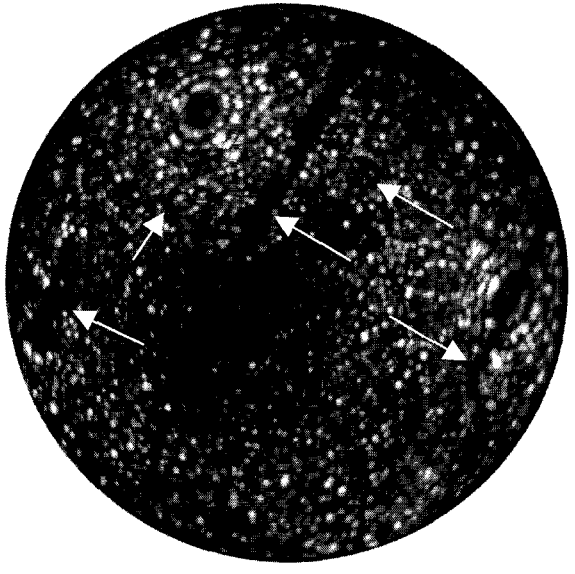
Recovery of a Magnetite Field Emission Tip



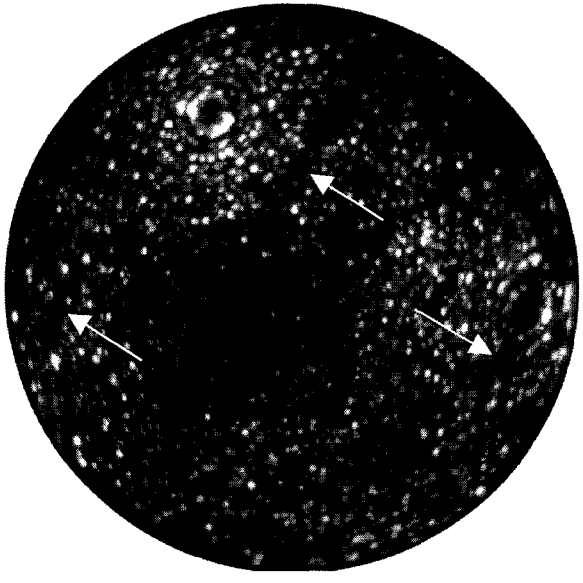
Surface after Field Evaporation

TEM of sample 031300E after resharpening with a Gatan ion mill with 5 keV Ar and gun tilt = 40°. a) Bright field image showing precipitates and bend contour. b) High magnification image of the tip and precipitates seen in FIM images. The line indicates the surface obtained after field evaporation.

Field Ion Micrographs of Magnetite LP204-1



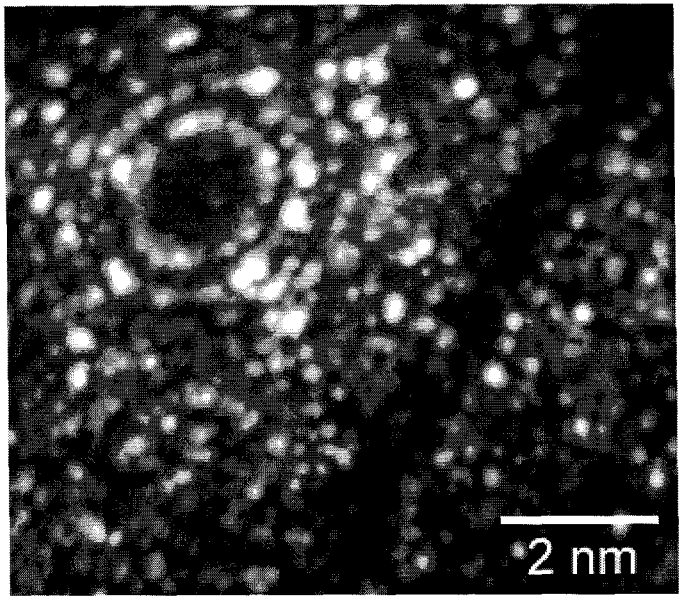
16 kV



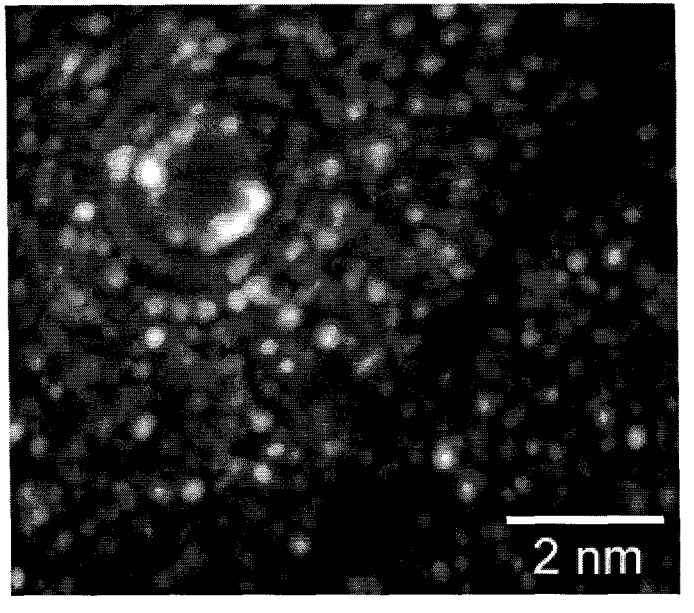
16.5 kV

Note the clarity of the {111} poles and the precipitates running directly through the pole at right of the images.

Six-fold Symmetry of the {111} Planes

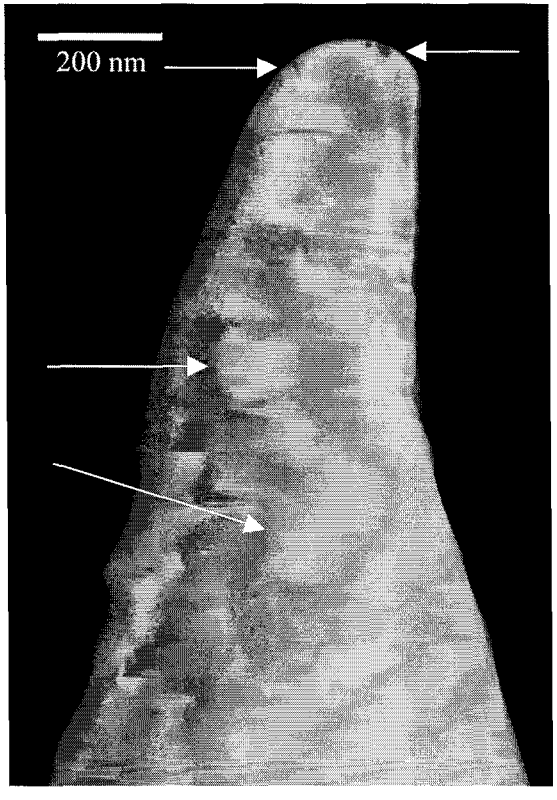


16 kV



16.5 kV

Post Evaporation TEM Analysis of Magnetite LP204-1

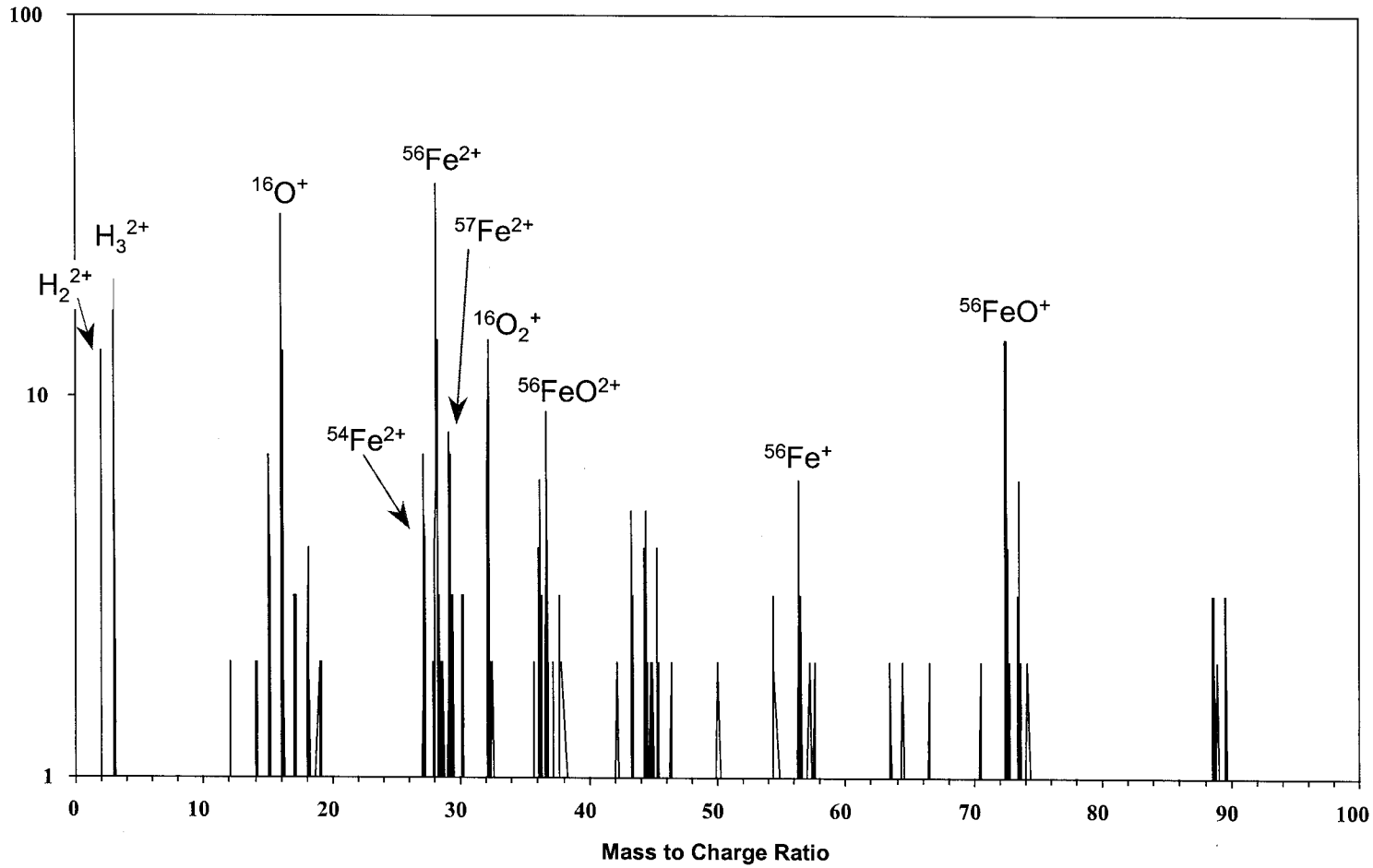


Precipitates seen in FIM images. The location of this surface is indicated on previous slide.

Disk-shaped precipitates normal to the beam.

Electron diffraction pattern indicates that this view is normal to the [100].

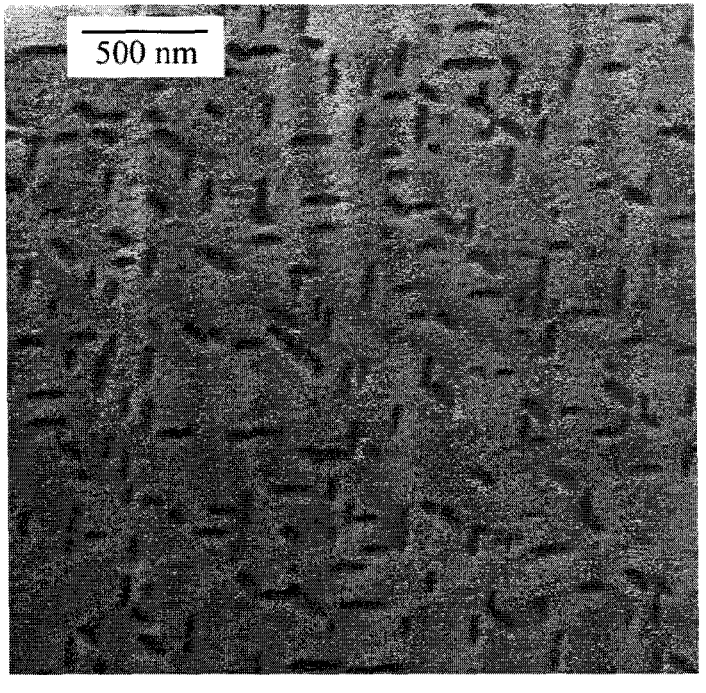
First Mass Spectra from a Geological Material using Atom Probe



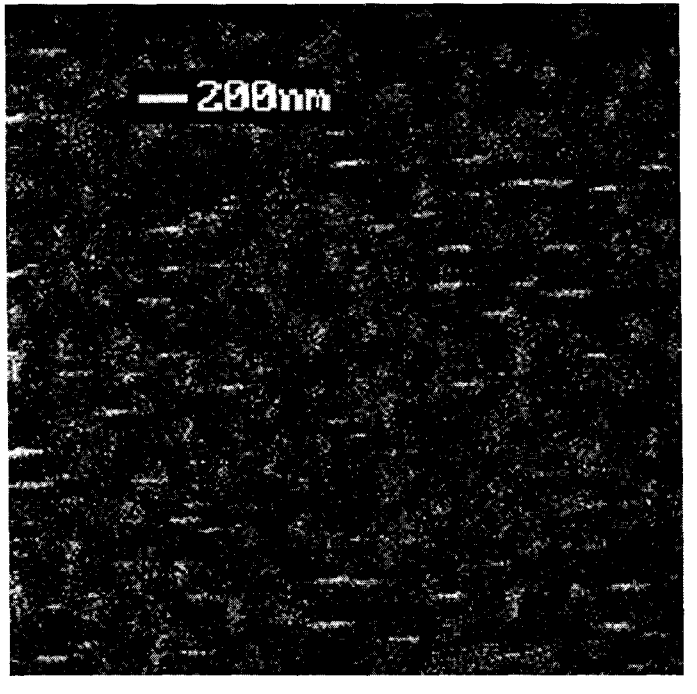
http://minileap.jpl.nasa.gov/

California Institute of Technology

Preliminary FIB-SIMS Imaging of Magnetite



Secondary Ion Image



Aluminum Map

FIB-SIMS Image of a Cross-Sectioned Spore

Conclusion

- First high quality FIM images of a bulk mineral.
- Preliminary APFIM analysis of magnetite:
  - Mass spectra have been obtained showing isotopes of Fe and O as well as FeO.
  - Singly and doubly charged ions are observed.
  - A preliminary mass spectrum taken from a precipitate contains both Mn and Al, as expected from previous analysis of magnetite LP204-1 by Sitzman, et al.
- Traditionally non-conductive samples ***CAN*** be imaged and analyzed using APFIM.
- FIB-SIMS can provide high-resolution chemical imaging of minerals.

Acknowledgements

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